

**UNIVERSITI TEKNOLOGI MARA**

**FEASIBILITY STUDY OF TiNb  
ALLOYS AS MEDICAL IMPLANT  
USING DIFFERENT PROCESSING  
TECHNIQUES**

**NUR HIDAYATUL NADHIRAH BT  
ELMI AZHAM SHAH**

**PhD**

**September 2019**

## ABSTRACT

The constant development of implant material and the search of biocompatible implant material of comparable mechanical properties towards the human bone have driven research on binary TiNb alloy. This study aims to develop  $\beta$ -rich TiNb alloy which is suitable for implant application and ultimately has the Young's modulus value which is close to the human bone. This study utilized elemental titanium (Ti) and Niobium (Nb) powders TiNb alloys with wt% of 10Nb, 25Nb, 40Nb and 45Nb. These alloys were produced utilizing three types of processing technique; arc melting, sintering in vacuum and sintering in argon atmosphere. All of the TiNb produced were compared in terms of phase constituent and mechanical properties. The phase arrangements and constituents of TiNb alloy were determined through Scanning Electron Microscopy (SEM) paired with Energy Dispersive X-ray (EDX) and X-ray Diffraction (XRD). As-melt and as-sintered Ti10Nb mainly consists of  $\alpha$ -phase Ti whereas Ti25Nb consists of mixtures of metastable phases meanwhile, Ti40Nb and Ti45Nb were rich in  $\beta$ -phase Ti. As sintered TiNb alloys had a better atomic arrangement in comparison to as melted samples based on the XRD patterns. Differential Scanning Calorimetry (DSC) was performed from the temperature of  $-50^{\circ}\text{C}$  to  $500^{\circ}\text{C}$ , clearly all of the samples does not show reversible  $\alpha'' \rightarrow \beta$  phase transformation. TiNb alloys produced via arc melting had the highest Vickers hardness value ranging from 414HV to 462HV followed by TiNb alloys produced via vacuum sintering with the range of 297HV to 362HV whereas TiNb alloy produced via argon gas sintering exhibited the lowest hardness value with the range of 173HV to 249HV. The Young's modulus obtained from the fabricated samples ranged from 24~30 GPa. These values are comparable to the Young's modulus of the human bone which is in the range of 20 GPa. Data obtained from the XRD and monotonic compression were used in linear regression to obtain a formula for estimating Young's modulus of TiNb alloy based on the phase constituents. Following the result obtained from phase and mechanical properties, the composition of Ti40Nb was selected for further prototype study of metallic bone staple. The samples were compacted and sintered in argon gas atmosphere followed by EDM wirecut to get the bone staple according to commercial size dimension. Based on the findings, Ti40Nb produced by sintering in argon atmosphere was fabricated into a metallic bone staple along with CpTi and stainless steel 316L grade. The bone staples underwent a four point bend test in order to measure its bending stiffness. The bending stiffness value obtained from the test on Ti40Nb produced via sintering in argon gas atmosphere was in the range of 7 N/mm. According to the fractured sample of Ti40Nb bone staple and the simulation made on four point bending, the most critical part of the bone staple is the connection between the bridge and the leg of the staple. Ti40Nb bone staple fracture exhibit microvoids coalescence dimples which indicates a ductile failure.

## ACKNOWLEDGEMENT

Thank you to Allah for providing me the opportunity to embark on this PhD journey and giving me strength and willpower, much needed to complete this long uphill road. I would like to express my special appreciation to Associate Professor Dr. Muhammad Hussain Ismail for his continuous guidance and unyielding support and performing his role as an excellent supervisor. Thanks to my co-supervisor Dr. Mahesh Kumar Talari who taught me the appropriate terms with regards to material science.

Also, thanks to my research mates who were with me exploring the beauty of Titanium based alloy and powder metallurgy. My appreciation also goes to my friends whom helped me through this journey and my siblings Fatin, Husna, Najmi and family who were always there for me. Not to forget, thank you to my best friend Hanis Athirah and my husband Muzhafar Azhar for their continuous moral support. Thank you to the supportive and understanding lab technicians of Mechanical Engineering and Applied Science faculty, Encik Rahimi, Encik Emy, Encik Mahmud, Encik Azrol, Encik Norazman and Encik Abul as they allowed me to conduct tests in their respective labs and gave me their full cooperation, guidance and help. Also, not to forget the general lab technicians of Chemical Engineering and general lab technician of Dentistry.

This thesis is dedicated to my beloved parents Mariam Haron and Elmi Azham Shah Marsom for their love, support and determination to educate me. This thesis is for the both of you. Alhamdulillah, thank you Allah for finally allowing me to complete this journey.

# TABLE OF CONTENTS

	<b>Page</b>
<b>CONFIRMATION BY PANEL OF EXAMINERS</b>	<b>ii</b>
<b>AUTHOR'S DECLARATION</b>	<b>iii</b>
<b>ABSTRACT</b>	<b>iv</b>
<b>ACKNOWLEDGEMENT</b>	<b>v</b>
<b>TABLE OF CONTENTS</b>	<b>vi</b>
<b>LIST OF TABLES</b>	<b>x</b>
<b>LIST OF FIGURES</b>	<b>xi</b>
<b>LIST OF PLATES</b>	<b>xvii</b>
<b>LIST OF ABBREVIATIONS</b>	<b>xix</b>
<b>CHAPTER ONE: INTRODUCTION</b>	<b>1</b>
1.1 Research Background	1
1.2 Problem Statement	2
1.3 Objectives	3
1.4 Scope of Study	4
1.5 Significance of Study	4
1.6 Thesis Outline	5
<b>CHAPTER TWO: LITERATURE REVIEW</b>	<b>6</b>
2.1 Introduction	6
2.2 Ti-based alloy as biomaterial	8
2.3 Ti-Nb Alloy	12
2.3.1 Phase diagram	12
2.3.2 Phase transformation in TiNb alloy and Differential Scanning Calorimetry	15
2.3.3 Microstructure and phase of TiNb	17
2.3.4 Mechanical properties of TiNb	23
2.4 Mechanical compatibility of metallic implant	28
2.5 Manufacturing of Ti alloy	30

# CHAPTER ONE

## INTRODUCTION

### 1.1 Research Background

Titanium (Ti) and its alloys are commercially used as implants and was first introduced to the biomedical field in 1940 [1] as it exhibits excellent corrosion resistance, strength and biocompatibility. Originally pure titanium and its alloys were intended for structural materials, such as airplane production purposes owing to its lightweight attribute and impressive strength. Categorized as a biomaterial, Ti is a nonviable material used in medical devices intended for contact with the living body [2] and often the preferred material used as implants in which this particular usage was revealed in 1952 by Professor Per-Ingvar Branemark M.D. [3]. He managed to insert tiny metal tubes into the bone of a rabbit to place microscope for the study of bone tissue. Ti was chosen owing to its attributes as a light, sturdy, non-corrosive metal. After several months, he attempted to take out the Ti sleeves from the rabbit's bones and was astonished to discover that he was incapable to extract them. The Ti had formed a permanent bond with the living bone and it did not appear to cause inflammation in the surrounding soft tissue, nor was it rejected by the living bone. He named this process of bone bonding to the metal as osseointegration. Ti may exist in  $\alpha$  phase,  $\beta$  phase or a combination of both with additional martensitic phases such as  $\alpha''$  and  $\omega$ , and Niobium (Nb) acts as a  $\beta$ -phase enhancer in Ti. In terms of implant material, the production of  $\beta$  phase rich alloy is very desirable as it has the Young modulus close to the human bone which is mainly attributed by the amount of  $\beta$ -phase in the alloy.

One of the most famous  $\beta$  type Ti alloy utilized in commercialize implant is NiTi. Like most  $\beta$ -phase Ti alloy, NiTi has two distinct features namely shape memory effect (SME) and superelasticity (SE) also known as psuedoelasticity (PE). Both of these characteristics are dependent to the phase transformation temperature which are strongly influenced by the ratio of Ni-Ti and also its processing technique [4]. TiNb, on the other hand, is among the many nickel (Ni)-free Ti alloys. TiNb offers an option free from Ni element which has a high probability of inducing allergy reaction in patients. There were not many studies on TiNb alloy and its development as a potential implant material. However, there were reports on the promising properties of TiNb alloy as